COMPARISON OF MEASURING INSTRUMENTS DESIGNED TO ELECTRICAL PROPERTIES ANALYSIS OF EPOXY RESINS WITH DIFFERENT FILLERS

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Abstract: Presented thesis is focused on studying electroinsulating epoxy resin-based sealings. It describes the chemical composition, production, properties and measuring methods of basic electric quantities of these materials. The aim of the thesis is to compare several sets of samples of composite epoxy resins with different kinds of micro-ground siliceous sand as a filling. The temperature and frequency dependence of relative permittivity, dissipation factor and volume resistivity are measured for given samples.

Keywords: Epoxy resins, thermosets, electrical insulation materials, dielectric, fillers, polyreaction, electrical properties, diagnostic techniques and measurement, permittivity, dissipation factor, resistivity

1. INTRODUCTION

Epoxy resins are relatively young plastics, first mentioned in the second half of the 19th century, but their production only developed after World War II.

These are synthetic polymers belonging to the thermosetting group. They are colorless to yellowish in normal conditions. Curing occurs most frequently with a chemical polyaddition reaction - no by-products occur. Cured products have very good electrical and dielectric properties, mechanical strength, especially shear strength, chemical resistance to water, acids and some solvents. They excel in adhesion to metallic, glass, ceramic, wood and other materials. Furthermore, epoxies are characterized by high toughness, low shrinkage and some good elasticity. [1]

2. SAMPLES OF CHS-EPODUR 494-1667

Samples were produced in laboratories of SYNPO a.s. Pardubice. CHS-EPODUR 494 1667 (CHS = unfilled potting compound, EPODUR = trade name, 494 = resin type, 1667 = hardener type) is a modified low molecular weight epoxy resin consisting of several components:

- components A - epoxy resin and polypropylene glycol,
- components B - hardener (tetrahydromethylphthalic anhydride),
- components C - accelerator (benzyldimethylamine)
- components D - flexibleizer (polyethylene glycol).

Next, a pigment paste (E-Pasta BF 135 M-BA) is added to the system. It consists of pigment and epoxy resin, determines the resulting color of the cured composite.

The last important ingredient is filler, added in the form of micro-milled quartz sands and significantly reduces the cost of the potting compound. Used fillers:

- set 1847: Silbond 126EST
- set 1848: Wollastonitmehl TREMIN 283-100EST
- set 1849: sand ST6 - Sklopísek Střeleč + Apyral 2E - Nabaltec
- set 1850: Silbond 126EST + Hydrafil Trefil 744-300EST
- set 1851: sand ST6 - Sklopísek Střeleč

The resulting 2 mm epoxy plates were cut into 80 x 80 mm square samples (for the Tettex measuring system) and 35 mm circular samples (for the Novocontrol measuring system). The cut was carried out using a water jet in cooperation with AWAC, spol. s r.o.

For measurement, it is important that the samples are planparal, therefore 5 samples with the smallest standard deviation were selected from the 10 square and circular samples for the purpose of the experiment.

3. MEASURING INSTRUMENTS

The Precision Oil and Solid Dielectric Analyzing System Tettex 2830/2831 is designed for measurement of liquid and solid insulating materials with a very low dielectric losses. The instrument works on the principle of a combined bridge-vector-meter and is capable of analyzing capacity and dissipation factor and DC-Resistivity with outstanding accuracy and stability. The instrument consists of a vector-meter bridge, temperature controller, DC and AC power supply and DC resistance measurement. The maximum operating voltage of the AC source is 2.5 kV, the frequency range is 40 - 65 Hz.

Measuring instrument for solid dielectrics - Tettex 2914 is connected to impedance analyzer - Tettex 2830/2831. Tettex 2914 is a three-electrode system consisting of a heated plate capacitor. The bottom plate (high voltage electrode) is on a solid base, the top plate (measuring and protective electrode) is adjustable in height by a hydraulic system that simultaneously compresses the electrodes against each other. The measuring electrode diameter is 49.5 mm, the protective electrode width is 10 mm, the distance between the measuring and protective electrodes is 1 mm. The electrodes are made of stainless steel. The maximum achievable electrode temperature is 200 °C. The maximum operating voltage is 2000 V. Other technical data can be found in [2][3].

Broadband Dielectric Spectrometer - Novocontrol Technologies Concept system 80 allows you to examine materials depending on temperature and frequency. The measuring system operates at 100 μV - 3 V AC, over a frequency range of 3 μHz to 40 MHz. The dissipation factor resolution is 10^{-5}. The highest accuracy is based on the high resolution of the Alpha-A frequency analyzer. The sample can be analyzed from temperature -160 °C to +400 °C thanks to the Quatro Cryosystem cryostat. The Active Sample Cell ZGS contains two electrodes - voltage and measuring. The voltage electrode has a diameter of 40 mm, measuring 20 mm. The maximum possible sample diameter is 40 mm. The measuring construction of instrument is made of stainless steel and the electrodes are gold plated.

4. DESCRIPTION OF THE MEASUREMENT

The aim of the experimental work was to compare the above mentioned measuring devices - Tettex 2830/2831 and Novocontrol Technologies - Concept system 80. Further analyze the electrical properties (relative permittivity, dissipation factor and volume resistivity) of the individual epoxy sets (1847 - 1851).

Measurement of relative permittivity and dissipation factor in dependence on temperature took place at working voltage 500 V, frequency 50 Hz and electrode pressure 5 N/cm^2 on impedance analyzer Tettex 2830/2831 in connection with measuring tool Tettex 2914 according to ČSN IEC 250 [4]. Samples were measured at temperature 23 ±1, 40, 55, 70, 85 and 100 °C, 5 times in succession. After reaching the desired temperatures, the sample was thermally stabilized for 30 min.

Frequency dependencies of relative permittivity, loss number, and dissipation factor were investigated on a Novocontrol Technologies dielectric impedance analyzer. Samples were measured at a voltage of 1 V, on a frequency range of 10 Hz to 1 MHz, and temperatures of 25 to 100 °C.

5. MEASUREMENT RESULTS

The dependencies of relative permittivity, loss number and dissipation factor on the frequency measured at 1 V, on the frequency range 10 Hz to 1 MHz and for temperatures from 25 to 100 °C are shown in Fig. 1, Fig. 2 and Fig. 3. The curves shown represent the mean value of the quantity obtained from the measurements on the five selected samples of the epoxy kit 1847. The measurement results confirm the polar character of the epoxy samples. It can be seen from the graphs that with decreasing frequency and increasing temperature, the components of complex permittivity and dissipation factor increase. The relative permittivity over the entire temperature range (25 - 100 °C) at a frequency of 10 Hz is equal to values in the range of 4.0 to 5.8 and at a frequency of 1 MHz it equals from 3.8 to 4.3. Identical graphical dependencies for other epoxy kits (1848, 1849, 1850, and 1851) are shown in [5].

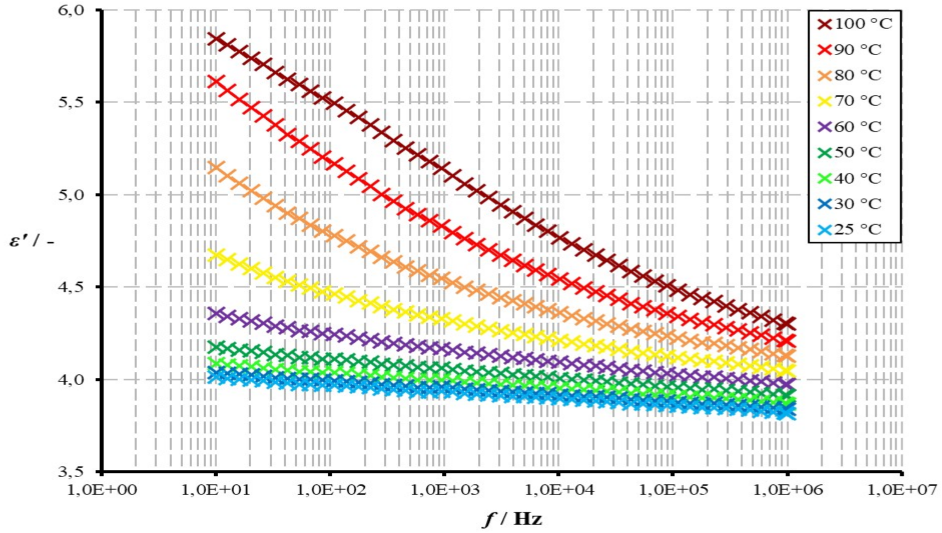

Fig. 1: Dependence of relative permittivity on frequency for given epoxy kit 1847, voltage set to 1 V

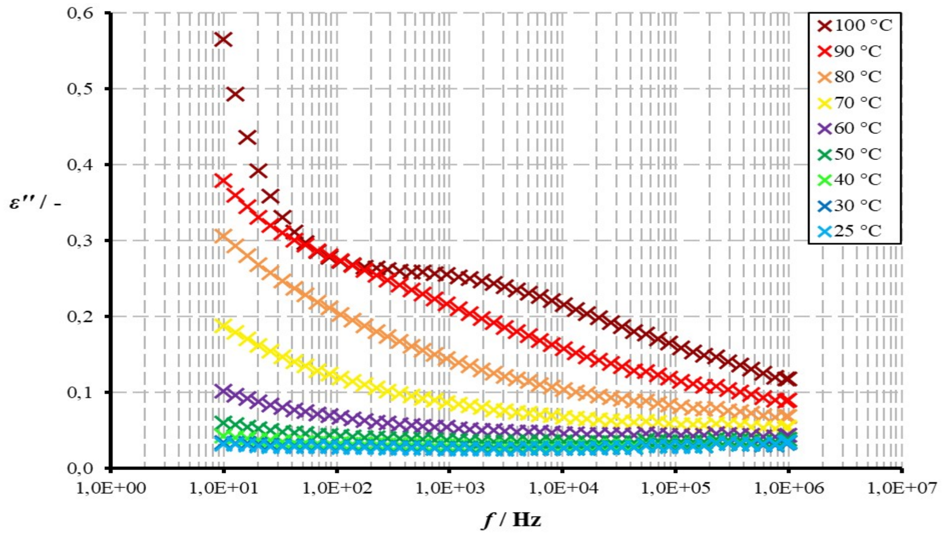

Fig. 2: Dependence of loss number on frequency for given epoxy kit 1847, voltage set to 1 V

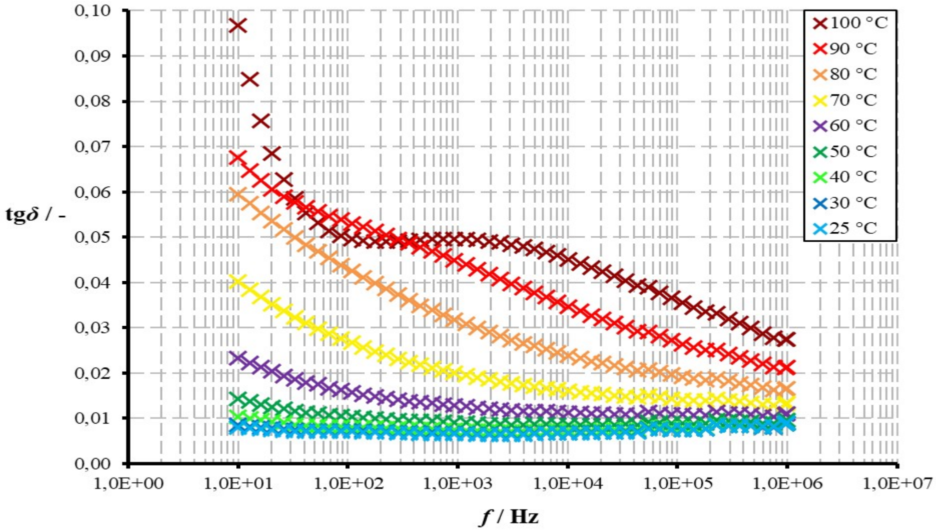

Fig. 3: Dependence of dissipation factor on frequency for given epoxy kit 1847, voltage set to 1 V

At a temperature of 90-100 °C, a change in the curve trend can be observed with respect to the loss number or dissipation factor (Fig. 2 and Fig. 3). The reason for this is that, in addition to conductivity losses, polarization losses are applied in the dielectric. Conductivity losses occur in all types of dielectrics, depending on the volume and surface conductivity of the dielectric. Polarization losses are affected by the type of polarization and are highly temperature and frequency dependent.

A comparison of the measuring instruments - the Tettex 2830/2831 dielectric analyzer and the Novocontrol Technologies impedance analyzer is shown in the graphs in Fig. 4 and Fig. 5. Temperature-dependent relative permittivity and dissipation factor measurements were performed at a working voltage of 500 V (for Tettex 2830/2831) and 1 V (for Novocontrol Technologies), at a mains frequency of 50 Hz and a temperature range of 25 - 100 °C. The real component of complex permittivity and dissipation factor are not dependent on the applied voltage (measured up to 2000 V). Without this finding, the measuring instruments could not be compared.

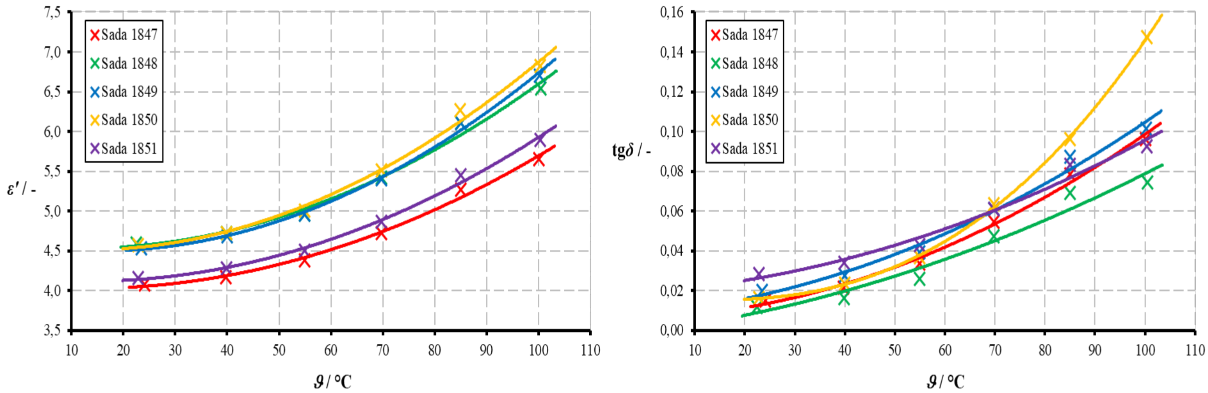

Fig. 4: Dependence of relative permittivity (left) and dissipation factor (right) on temperature
at 500 V and 50 Hz - Tettex 2830/2831

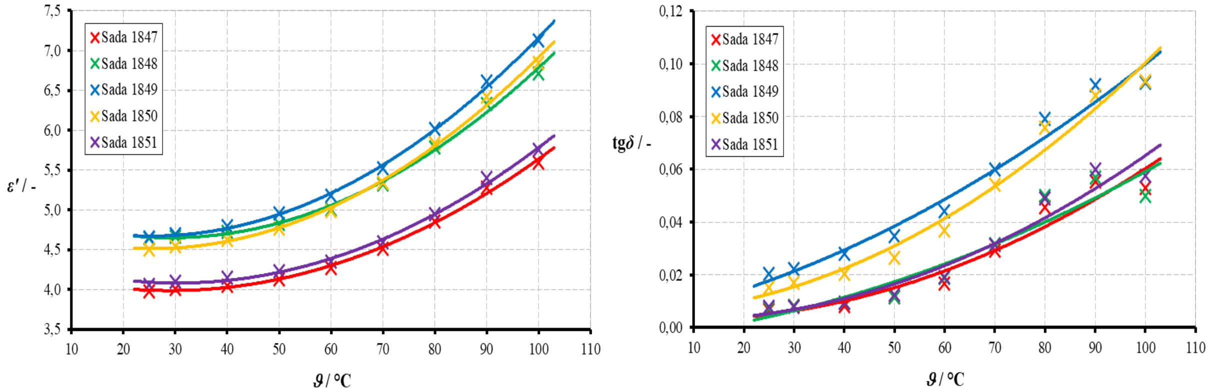

Fig. 5: Dependence of relative permittivity (left) and dissipation factor (right) on temperature at 1 V and 50 Hz - Novocontrol Technologies

6. CONCLUSION

In this frequency range, the lowest relative permittivity values of epoxy resin with filler Silbond 126EST (set 1847) and the highest values are epoxies with added fillers ST6 from the Czech company Sklopísek Střeleč and Apyral 2E from the German company Nabaltec (set 1849). It follows from the experimental work that all the analyzed epoxy materials exhibit very good electrical insulation properties for the given field of application. This corresponds to the calculated one-minute polarization indices, which are in the range of 2.8 - 3.2. The best electrical properties (low dissipation factor) are achieved with epoxy resins with added Wollastonitmehl TREMIN-283-100EST filler (set 1848) and conversely, the worst results are measured for epoxy with fillers Silbond 126EST and Hydrafil Trefil 744-300EST (set 1850). The most used in technical practice are epoxy resins with added micronised quartz sand ST6 from Sklopísek Střeleč (set 1851). The measured data of these epoxy samples reach average values.

The experiment also includes a comparison of the above-mentioned devices - the Tettex 2830/2831 dielectric analyzer and the Novocontrol Technologies - Concept system 80 impedance analyzer. Measured data from both devices can be considered as appropriate.

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